Typology of limestones from Cameroon for fresh water remineralization: morphology, mineralogy and geochemistry

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Abstract: Eight (8) limestone samples were collected from three regions of Cameroon (South, Littoral, and North) for the treatment of aggressive water through remineralization. The samples were analyzed to determine which of them would be most effective for the treatment. The tests carried out included DRX, IRTF, X-ray fluorescence, MEB-EDX, and ATG-DSC. It was found that all the rocks were made of calcite, with peaks of value d=3.035, 2.285, 2.095, 1.913, 1.875, 2.495, and 3.86 for XRD and bands 2515 cm^{-1}, 1407 cm^{-1}, 873 cm^{-1}, and 712 cm^{-1} for IRTF. However, a larger percentage of calcite was found in the marbles collected from the North. Some of the samples contained compounds that made them unsuitable for water treatment. he main criteria for selecting the best limestone were the calcite content of the rocks and its overall composition (the presence of non-reactive or harmful elements). Consequently, white marble was found to be the most suited rock for the desired treatment.

Keywords: Limestone; Aggressive water; Mineralogical analysis; Morphology

1. Introduction

Cameroon boasts a significant geological potential for various minerals, making it an attractive economic destination. Among the valuable deposits in the country are iron, bauxite, diamond, limestone, rutile, cobalt, and nickel (Lemougna *et al.,* 2023). Although limestone is widely distributed in Cameroon (Gweth, 2001; Bahiya *et al.*, 2018; Bisse *et al.,* 2018; Lyonga *et al.,* 2022), it is underutilized. The country relies on it mostly as an essential component in the manufacturing of cement, tiles, and slaked lime (Toumba et Wakponou, 2014; Yamb *et al.,* 2016; Shahrabadi *et al*., 2017). However, it has other applications in adhesives, paints, inks, stationery, food, and pharmaceutical industries (Oates,1998; Geyssant, 2001). On the other hand, it is evident that most water in tropical regions, particularly in Cameroon, is aggressive, as reported by authors (Guilleret *et al.*, 1990,

Kamgaing *et al.*, 2008, and Nguemtue *et al.*, 2020). This is detrimental to the distribution network and indirectly affects human health. Unfortunately, little attention is given to this aspect. After water clarification, manufacturers inject some soda, but this treatment is ineffective, lacking the necessary persistence until consumption. Notably, remineralization is considered the best treatment for aggressive water. This enriches the water with vital minerals, essential for human health, while protecting the distribution infrastructure, as confirmed by Degrémont, 2005. Limestone is a commonly used material for the remineralization process, particularly in regions where desalinated or reverse osmosis water is utilized (Ruggieri *et al.*, 2008; Shemer *et al.*, 2013; Al-Ghamdi, 2017). Limestone and marble are primarily made of calcium carbonate, but not entirely, and there are three polymorphs: calcite, the most abundant and widespread variety (Smyth, 1997), aragonite which is metastable and vaterite, the least thermodynamically stable (Ni and Ratner, 2008; Singh *et al.*,2016). Studies on their use are also numerous. Indeed, their applications are diverse, such as flue gas desulfurization process, neutralization of acid waters, building materials and roading, biomineralization (Santoro et *al.*, 1987; Gökay and Gundogdu, 2008; Ni and Ratner, 2008; Siagi and Mbarawa, 2009; Carletti *et al.*, 2013; Kapil and Bhattacharyya, 2017; Szlugaj and Galos, 2021; Rudenko *et al.*, 2023). Their chemical composition varies with the region in which they are collected (Pokrovsky *et al.*, 2005). Furthermore, their exploitation and use can cause damage to human health and the environment (Toundou *et al.*, 2017; Oyeyemi *et al.*, 2019). Thus, rock characterization plays an essential role by providing scientific information to aid in decision-making concerning the material's use. The primary objective of this study is to make a preferable selection for water treatment, specifically the correction of water aggressiveness through remineralization by discriminatory choices. For this purpose, eight (8) samples were retrieved from three different regions and examined by techniques including DRX, IRTF, X-ray fluorescence, MEB-EDX, ATG-DSC. Various studies (Shih *et al.*, 2000; Siagi and Mbarawa, 2007; Ruggieri *et al.*, 2008) have conducted similar research aiming to select the best limestone using textural and calcium carbonate composition as discrimination criteria. Conversely, our study emphasizes the use of mineralogical and textural parameters to achieve favorable remineralization outcomes.

2.   Geological and geographical setting :

The sampling locations of this study are shown in Figure 1. They are located in Cameroon and include the South (Mintom), Littoral (Kompina and Logbadjeck), and North (Figuil) regions. The area of Mintom is made up of metasedimentary and volcano-sedimentary rocks with ferralitic soils in the south and forested vegetation (Caron *et al.*, 2010). Kompina and Logbadjeck are situated in the Littoral region, which is part of the West-Central Coastal Province, with clayey ferralitic soils that influence the quality of collected limestones (Bukalo, 2018). The North region is mainly composed of cretaceous deposits with savannah vegetation, and the sample collected in Figuil is made up of marbles resulting from the metamorphism of limestone.

3.   Methods :

In this study, eight samples designated as Ca1 to Ca8 were collected to conduct chemical, mineral, and morphological analysis. To obtain the necessary data, the samples were crushed into a powder form and subjected to qualitative X-Ray Diffraction (XRD) analysis. XRD patterns were captured using the PANalytical X'Pert PRO MPD diffractometer, running at a voltage of 45 kV and an intensity of 30 mA, under 2 theta with Co Kα radiation of 1.542 Å. Furthermore, elemental analysis was carried out using the Nexus Thermofisher FTIR equipment to identify chemical functional groups. The energy dispersive X-ray fluorescence spectrometer (Pocket III) was utilized to analyze major elements. To supplement

this, EDX analysis was conducted using a HITACHI model S-4500 instrument, which allowed for morphological imaging of the samples with nanometer resolution. Additionally, thermal analysis was conducted using the TGA/DSC Q20 Module instrument. X'pert Highscore and Origin pro 9 software were used to analyze both XRD and FTIR spectra.
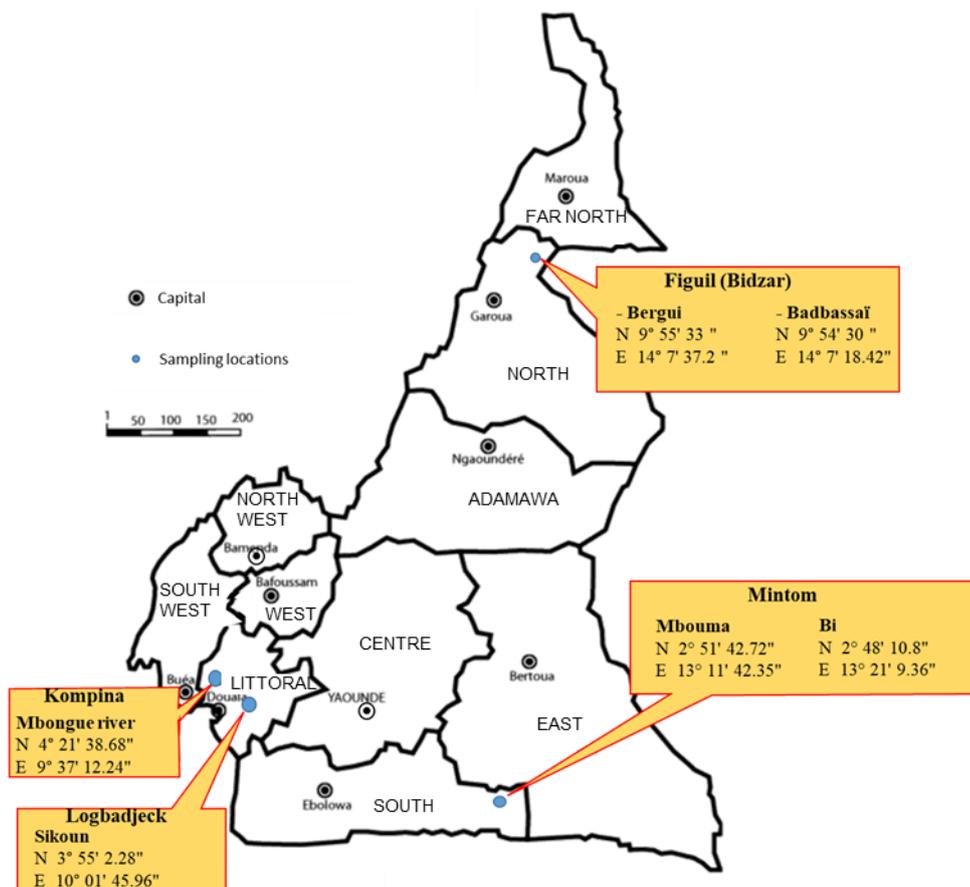
Figure 1. Map showing the locations of samples collection

4. Results and discussion

4.1. Mineralogy of limestones samples

Analysis of samples through XRD revealed that calcite is the major and the dominant mineral. Figure 2 shows the X-ray patterns of samples. All these samples are indeed made of calcium carbonate in the form of calcite. The values of d = 3.035; 2.285; 2.095; 1.913; 1.875; 2.495; 3.86 confirm this. These results corroborate those of the analyzes of the samples made by Gunasekaran and Anbalagan, 2007, and Bawa et *al.*, 2021. Analysis with FTIR has been perfomed to find superficial chemical groups. Samples sprectra are presented in Figure 3. We also deduced that samples are made from calcite. Indeed, the characteristic peaks are present; it is a weak band around 2515 cm^{-1}, an intense band around 1407 cm^{-1}, a thin and medium band around 873 cm^{-1} and a thin and weak band around 712 cm^{-1}. Kaolinite ($Al_2O_3,2SiO_2,2H_2O$) have been also detected at 663 cm^{-1} for Ca4, around 1010 cm^{-1} for Ca7 and Ca8 rocks which were collected in the same region, and at 3631 cm^{-1} for Ca6. Previous studies have shown that Ca3 contains dolomite $CaMg(CO_3)_2$ which dissolves more slowly than calcite in solution (Ruggieri et *al.*, 2008, Nguemtue et *al.*, 2020). This is also found in Ca3 with 1803 cm^{-1} and 2524 cm^{-1} even if these peaks are very low. We further observed a characteristic peak of quartz (SiO_2) at 1041 cm^{-1} for Ca5 and 1054 cm^{-1} for Ca6. Ruggieri et *al.*, 2008, affirmed that the presence of non-reactive elements as quartz contributes to the increase of suspended matter in the water, and this is the case for quartz.
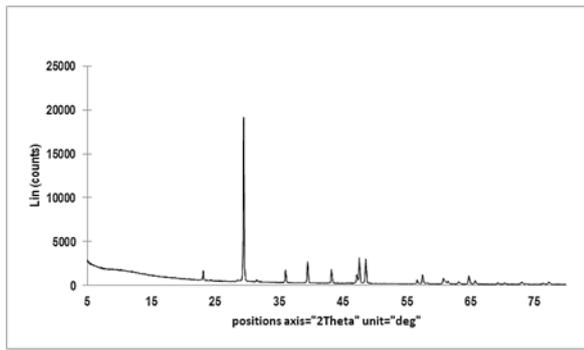
Ca1 (Marble white veins)
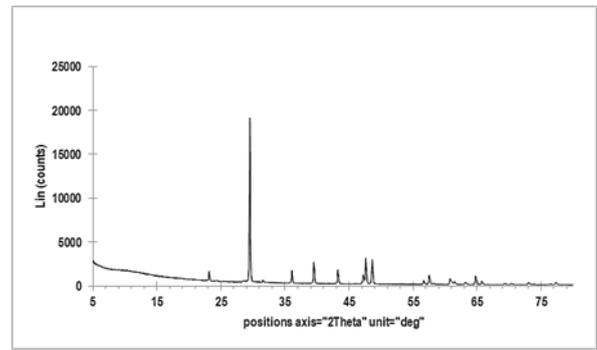
Ca2 (Marble green veins)
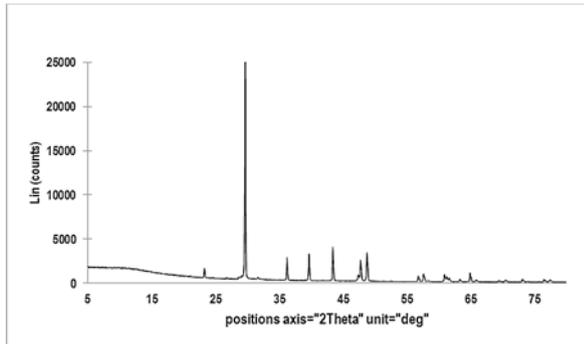
Ca3 (Marble orange veins)
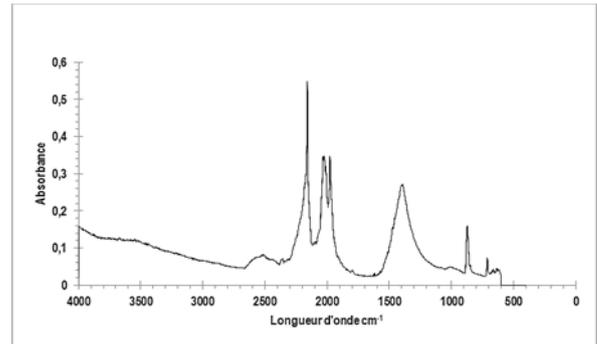
Ca4 (Marble black veins)
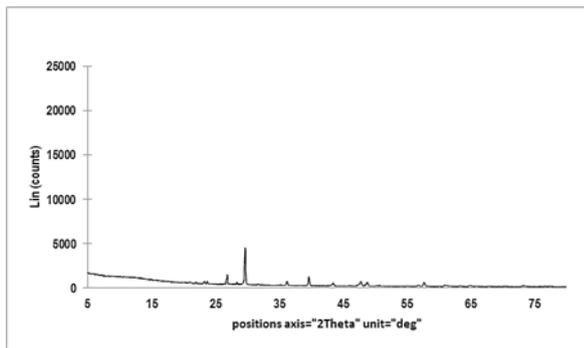
Ca6 (Logbadjeck limestone)
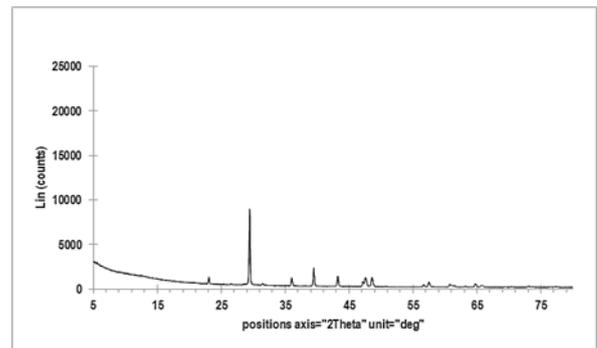
Ca5 (Kompina limestone)
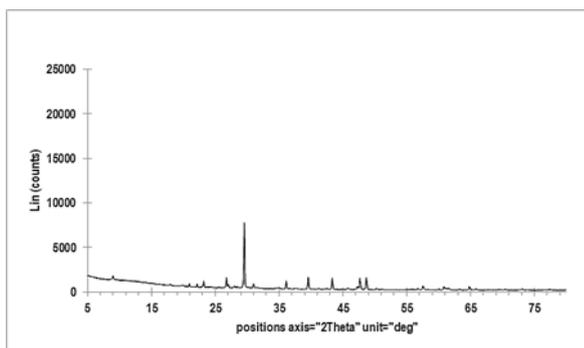
Ca7 (Mintom 1 limestone)
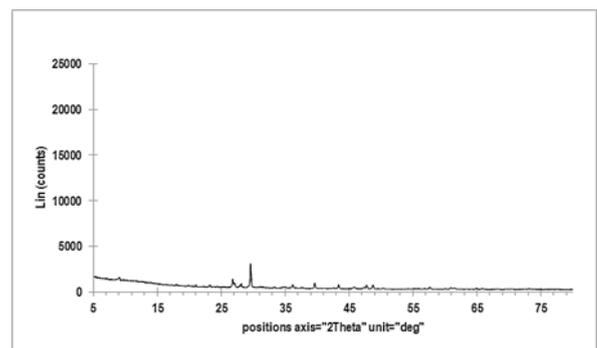
Ca8 (Mintom 2 limestone)

Figure 2. Samples XRD patterns

Others non-reactive elements as clays (kaolinite in this case) reduce dissolution capacity of calcite by coating it. Thus, the choice of the limestone with a minimum of these element content is considered. Some peaks appeared between 1900 cm^{-1} and 2500 cm^{-1} which corresponds impurities. They are found in Ca2, Ca4 and Ca8 rocks. This could be organic matter, as carbon, or other minerals, such as magnesium carbonate. As our goal is to find best limestone(s) and considering the above, it will not be

interesting to use them. Although the other rocks have low impurities, the shape of the peaks also provides information on the quality of their crystallization. The widened peak around 1400 cm^{-1} for rocks Ca3, Ca6 and Ca8 showed a non-perfect crystallization.
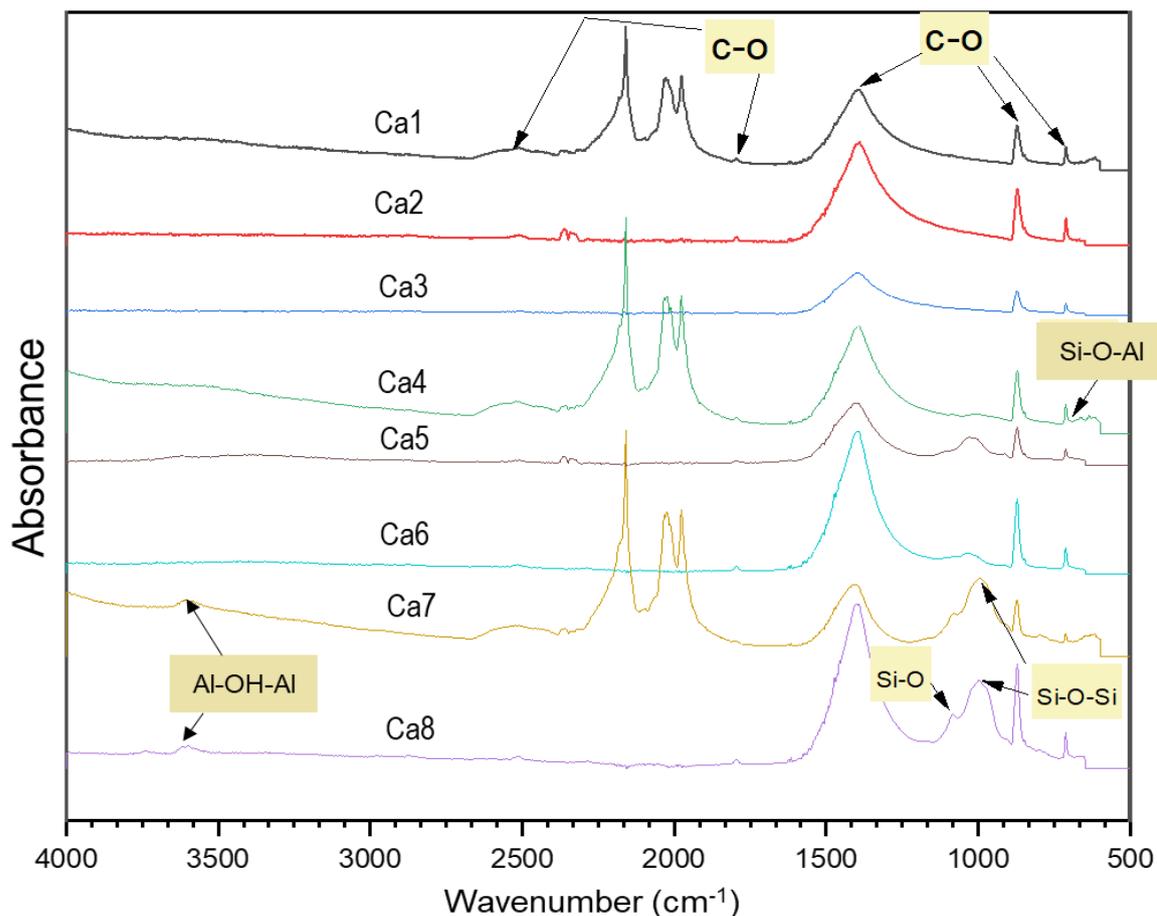
Figure 3. FTIR spectras of limestones samples

4.2. Geochimistry

X-ray fluorescence analysis of rocks allowed to give their chemical composition (Table 1). It should be noted that the marbles are quantitatively more composed of calcite, with a very important percentage of calcium. Some other elements could also be determined, in this case titanium and iron which are present in all rocks and in significant quantities. It is common to find in ores these elements which are found as oxides: TiO_2 and Fe_2O_3. These titanium and iron values are higher for Ca6, Ca5, C7 and Ca8 rocks. This is confirmed in most of these cases through the EDX spectra. But we also notice a fairly high iron value for Ca3 and Ca4, which could be the origin of Ca3 veins orange color. The green color of Ca2 would come from chlorite $(Fe,Mg,Al)_6(Si,Al)_4O_{10}(OH)_8$. Trace elements, that is, Co, Ni, Cu, Zn, Zr, Nb, As, K, Ta, Rb, Sr, In, are minor in rocks, they are generally found in limestones (Bawa et *al.*, 2021) as oxides.

Calcium content given here is higher for marbles Ca1, Ca2, Ca3, Ca4 than in others limestones. This is respectively an average calcium content of 53.12% for Ca1, 43.92% for Ca2, 46.09% for Ca3, and 44.63% for Ca4. This corresponds to a calcite content around 100% and even more, if we consider that the percentage of calcium in $CaCO_3$ is 40%. Note that these values can be considered approximate due to the method of determination. However, the most important information drawn from this is that the marbles are essentially made of calcite. The value of the loss on ignition would confirm the existence of the impurities seen on the FTIR spectra, for Ca4 and Ca8, whose values are the higher.

Table 1. X-ray fluorescence chemical characterization of limestones samples.

Sample	Ca(%)	Ti (%)	Fe (%)	Co (%)	Ni (%)	Cu (%)	Zn (%)	Zr (%)
Ca1	53.13	6.2E-02	3.1E-02	4.2E-03	—	1.8E-04	—	—
Ca2	43.92	6.9E-03	5.3E-02	—	1.2E-02	5.5E-04	—	5.5E-02
Ca3	46.09	7.1E-02	8.7E-02	1.5E-02	—	—	1.3E-04	—
Ca4	44.63	7.3E-02	1.3E-03	—	—	—	—	—
Ca5	20.06	0.10	0.52	—	—	—	5.2E-04	4.4E-02
Ca6	12.94	1.5E-03	1.47	—	1.3E-03	3.4E-04	1.6E-03	3.1E-02
Ca7	14.92	0.12	1.49	—	1.5E-04	1.2E-03	3.4E-03	1.4E-02
Ca8	11.78	0.10	2.47	—	—	—	4.6E-03	—

Sample	Nb (%)	As (%)	K (%)	Ta (%)	Rb (%)	Sr (%)	In (ppm)	LOI*(%)
Ca1	—	—	—	2.0E-05	—	1.8E-02	6.76	13.41
Ca2	6.8E-03	—	5.3E-04	—	2.5E-03	0.10	0.19	11.95
Ca3	—	4.3E-04	—	—	2.5E-04	0.04	5.31	13.00
Ca4	8.1E-04	4.7E-04	—	3.7E-05	1.3E-04	1.8E-02	6.85	21.36
Ca5	1.0E-04	—	—	1.1E-04	2.7E-03	6.3E-02	4.89	16.13
Ca6	3.4E-03	5.5E-04	1.4E-02	5.2E-04	3.1E-03	5.6E-02	20.85	11.86
Ca7	—	5.2E-04	1.79	1.9E-03	5.9E-03	0.10	2.78	17.46
Ca8	1.6E-03	—	1.82	7.5E-04	5.4E-03	5.6E-02	5.06	19.13

*LOI : loss on ignition

EDX microanalysis complements X-ray fluorescence analysis (Figure 4). The X-ray spectra taken for one point in each rock revealed more elements: Na, K, Mg, Al, Si, Ti, Fe, P, S, Sc, and Os. Most of these elements are actually found as oxides (Na_2O, K_2O, MgO, Al_2O_3, TiO_2, Fe_2O_3, P_2O_5). Many authors also found most of these elements during their analyses (Gunasekaran and Anbalagan, 2007; Ruggieri et al., 2008; Biskri et al., 2016). From marbles Ca4 seems to have more impurity. We note the presence of iron Fe in the other limestones. These spectra confirm that limestone is predominant in marbles Ca1, Ca2, Ca3, and Ca4, but this last one contains Aluminum and Iron. Although Aluminum content is very low, using of a limestone containing it is not suitable for water treatment. On the other hand, Iron leads a reddish color that will still need to be treated afterwards. The spectra of Ca6, Ca5, Ca7 and Ca8, also confirmed their low calcite content and the presence of some elements that are not recommended for human health, such as osmium (Smith et al., 1974).

4.3. Morphological analysis

The analysis of the morphology of our samples was carried out, and SEM images of selected samples Ca1, Ca5, Ca6, and Ca7, at a scale of 10 μm, are presented in Figure 4, considering their geological origin. Because the rocks were roughly crushed, they consist of coexisting large and small particles. The crystalline faces of calcite in marbles Ca1, Ca2, Ca3, and Ca4 are clearly visible, and these carbonates are aggregates that consist of rhombohedral amphibolite facies, which are more compact in marbles. From examining the images, we noticed that there were four metamorphic rocks, namely Ca1, Ca2, Ca3, Ca4, Ca7, and Ca8, and two sedimentary rocks, namely Ca6 and Ca5. While rocks Ca7 and Ca8 present flattened agglomerates facies, Ca6 and Ca5 have diverse morphologies.
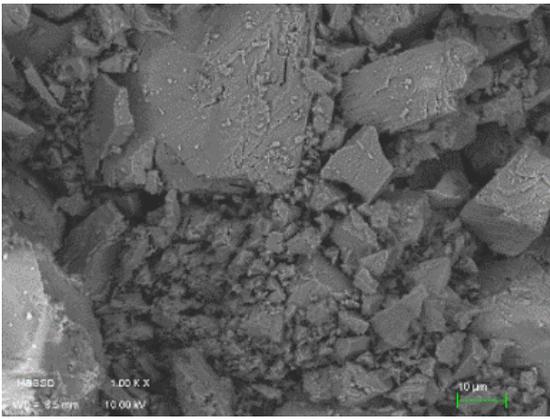
Ca1
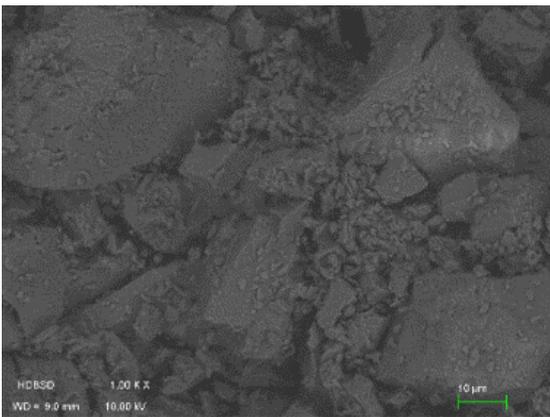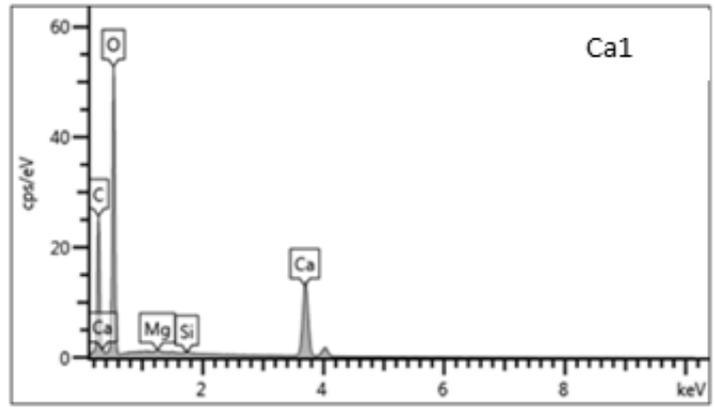
Ca2
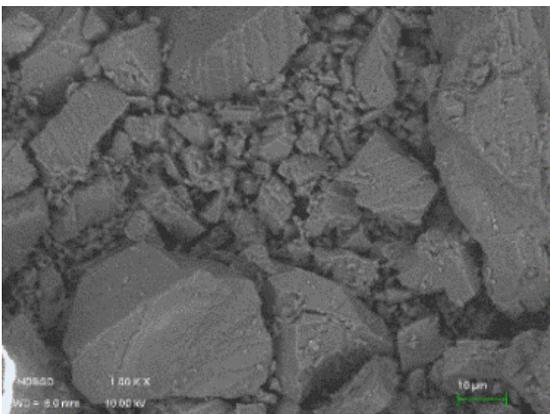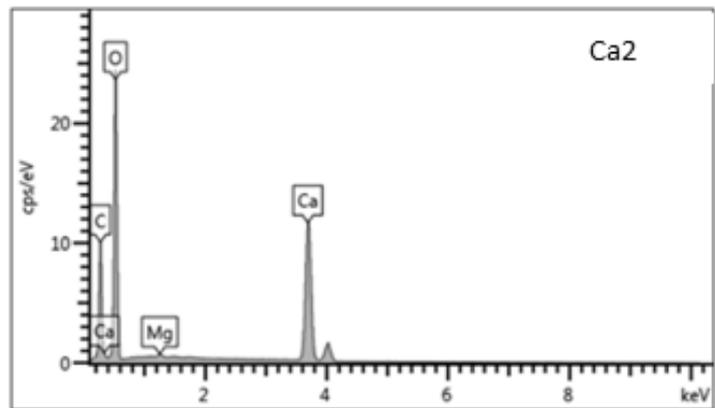
Ca3
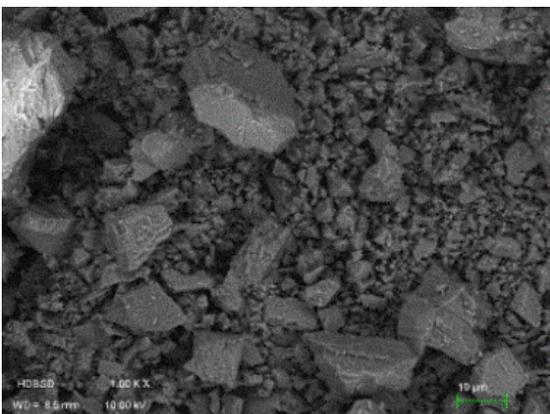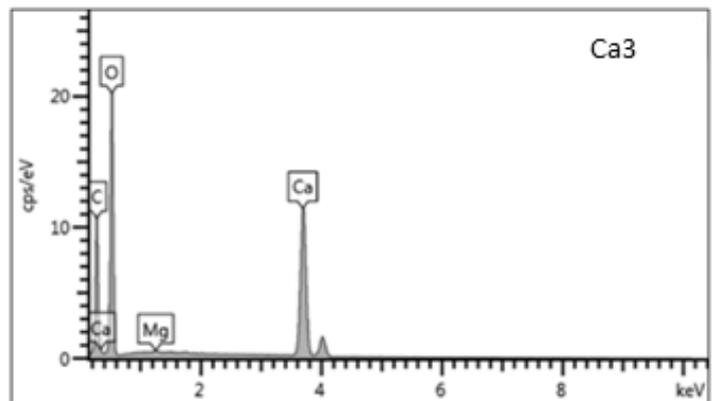
Ca4
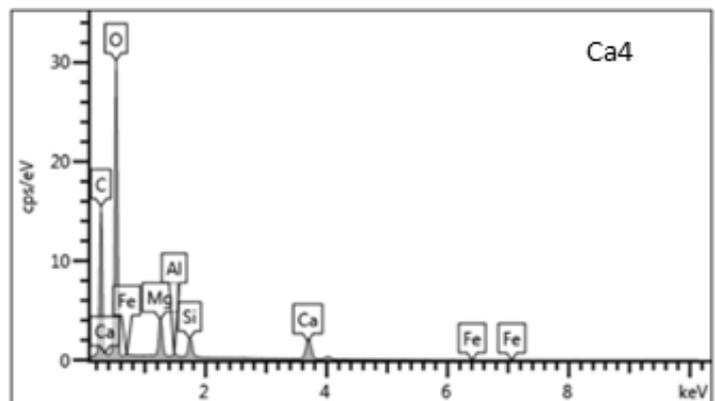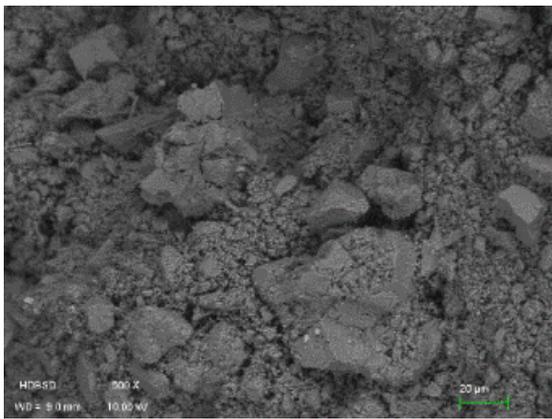
Ca5
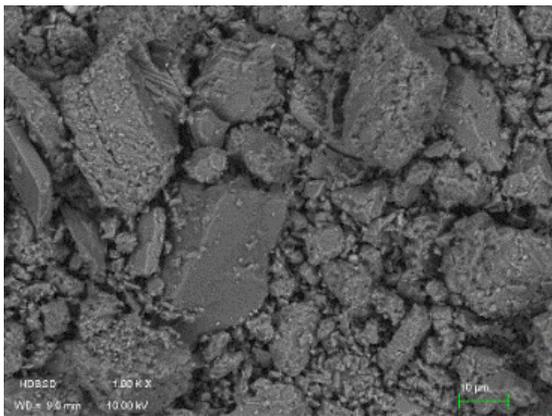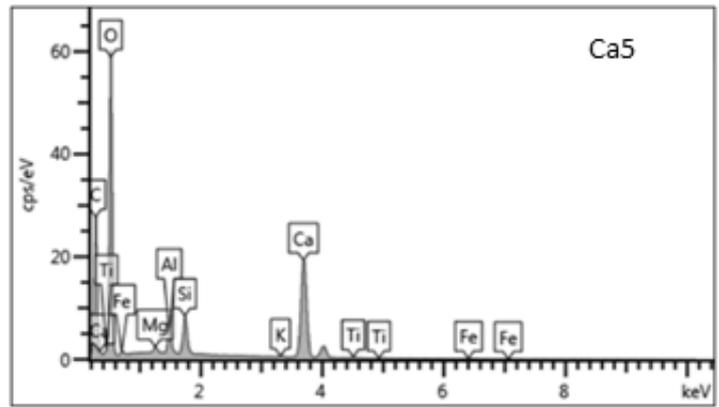
Ca6
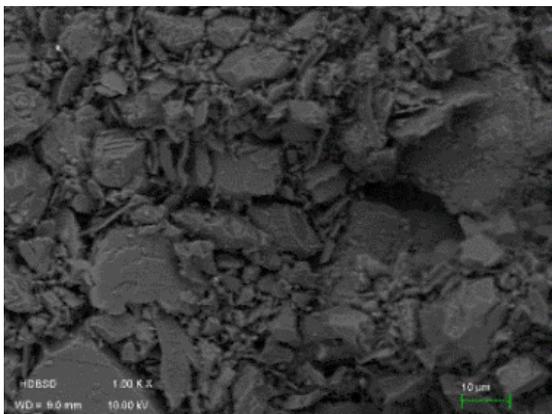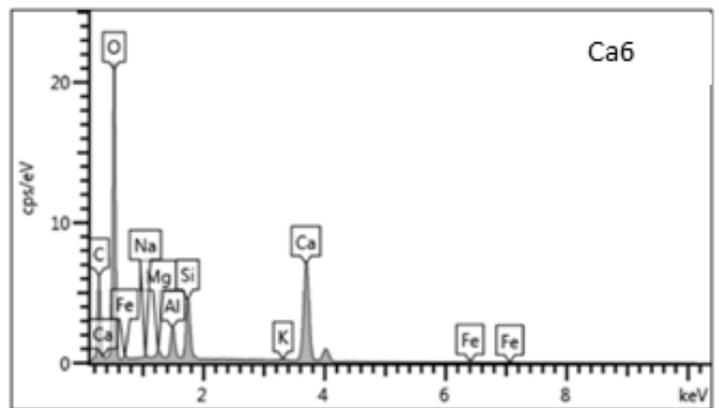
Ca7
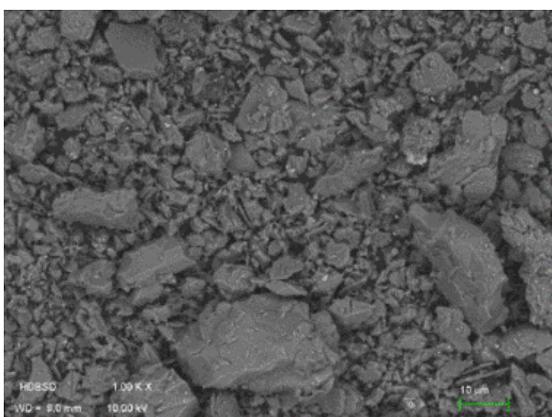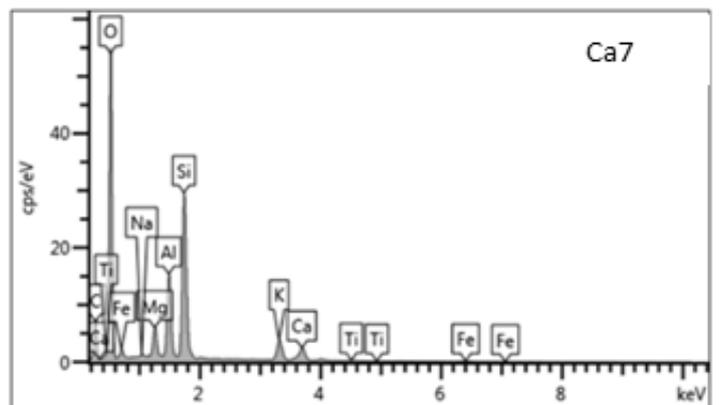
Ca8
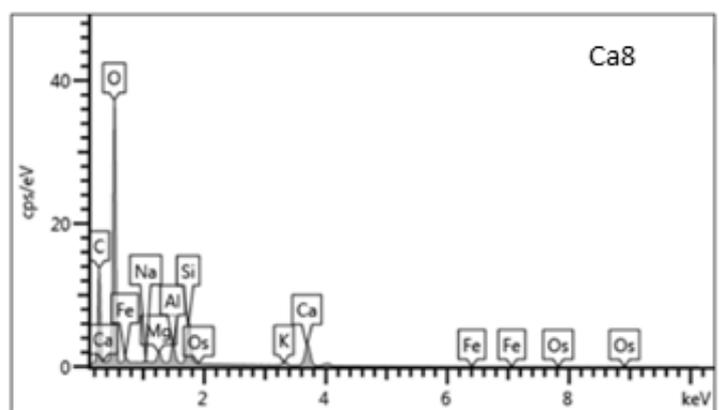
Figure 4. SEM images and some choosen correspondant EDX spectras for limestones samples

As they are sedimentary of origin, they are likely to be crumbled, hence their varied morphology. This is especially noticeable in Ca6 limestone, which is a Silurian fossiliferous rock containing fossils of Lamellibranch mollusks (bivalves), Gastropods, and Brachiopods. These types of fossils can also be seen in Ca5, but the dimensions are reduced, which can make it difficult to differentiate from calcareous material.

4.4. Thermal stability

The TGA-DSC curves (Figure 5) show a calcite decomposition phenomenon characterized by a sudden decrease of the thermogravimetric analysis (TGA) curves at temperatures from 620° to 800°C, and its rise until around 820°C. The differential scanning calorimetry (DSC) curves obtained showed a single endothermic peak around 785°C for all the samples, with the exception of Ca8 which deviates slightly from this value with 749.58°C. Their shape indicates that the decomposition process is initially very slow, and brutal after. Karunadasa et al., 2019, who also worked on calcite (purity > 99.9%) found that this peak was at 790°C, which is very close to our values. This analysis confirms again all our samples contain calcite. This decomposition equation corresponds to:

$$CaCO_{3\,(s)} \xrightarrow{\Delta} CaO_{(s)} + CO_2 \qquad \textbf{Eqn. 1}$$
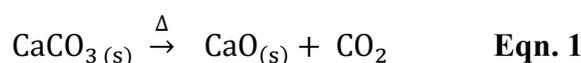
This reaction is pseudomorphic and topotactic (Rodriguez-Navarro et al., 2009). It begins with the formation of a mesoporous structure consisting of up to four sets of rod-shaped CaO nanocrystals oriented on each rhombohedral cleavage face of the pseudomorphic calcite.
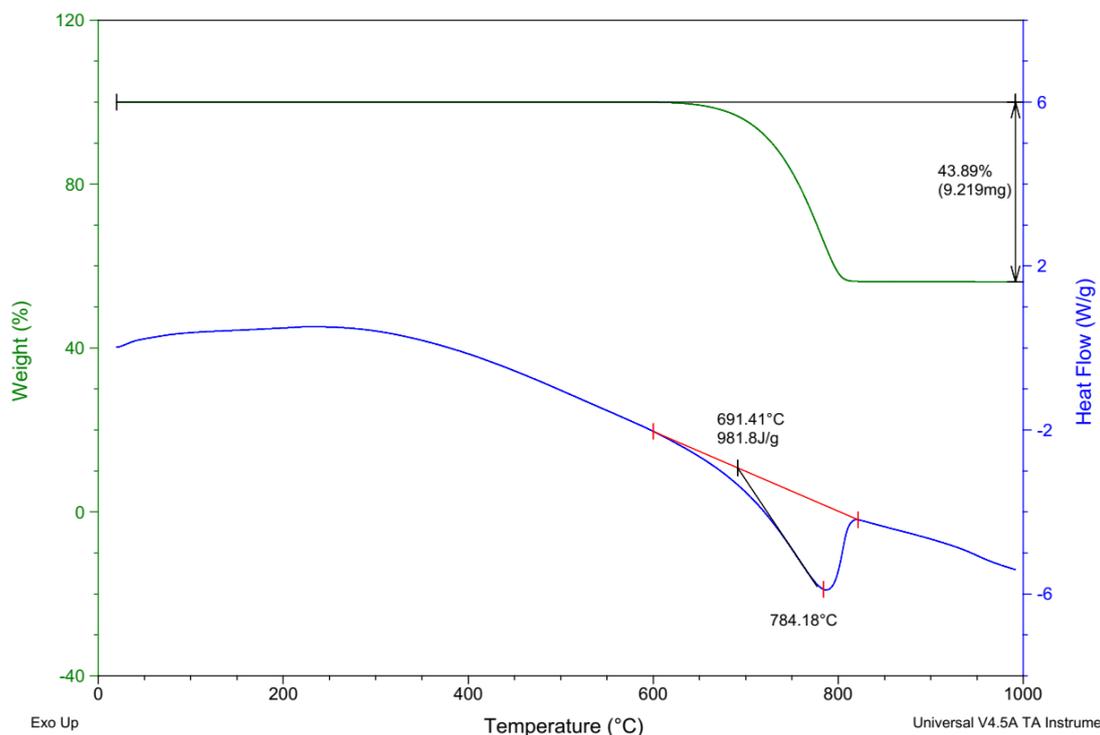
Figure 5. TGA and DSC curves for sample Ca1

Mass losses related to the release of carbon dioxide are almost the same for marbles that is, around 44%. This also corresponds to that of Karunadasa et al., 2019. The other limestones have values from 41.86% to 24.12%. From this mass loss, the approximate percentage of $CaCO_3$ can be deduced. Indeed, considering equation (1) above, and knowing that each curve gives informations about initial and after

loss mass, the CO_2 loss corresponds exactly to the mass of $CaCO_3$ in the sample. Their ratio is $M_{CaCO3}/M_{CO2} = 100/44$. Thus, the mass m_1 of $CaCO_3$ in the sample will be:

$$m_1 = m_{CO2} \times 100/44$$

And $CaCO_3$ content will be $\qquad p(\%) = (m_1 \times 100)/m_{sample}$

We deduced that $\qquad\qquad\quad p(\%) = (m_1 \times 10000)/44 m_{sample} \qquad$ **Eqn. 2**

These percentages were calculated (Table 2) and discussed. These values are close to 100% or greater than it, for Ca5 and marbles, and that confirm the deduction made from X-ray fluorescence analysis. The calcite content of Ca7 is higher than that of Ca8. When collected, this rock seemed to in the process of decomposition.

From the DSC curves we determined the decomposition temperature of the samples, and the specific heats. The temperatures are not the same, even if it is the same thermal decomposition reaction. This is explained by the presence of impurities in the samples. Some of these impurities have been detected in FTIR spectra. As for the specific heats, we realize the marbles have values ranging from 981 to 1065 J/g. For the others, the values are quite variable, certainly due to the diverse mineral composition of these rocks.

Table 2. Data from curves and Calcium carbonate content

Sample	Mass of analyzed sample	Mass loss (in CO_2)	Calcium carbonate content (%)	Decomposition Temperature (°C)	Mass specific heat of decomposition (J/g)
Ca1	21.008	9.21	99.73	784.18	981.8
Ca2	34.989	15.43	100.22	802.26	990.3
Ca3	18.827	8.32	100.52	774.25	1065
Ca4	18.859	8.12	97.85	778.46	986.4
Ca5	23.832	9.14	87.16	787.88	943.4
Ca6	20.367	7.38	82.43	776.15	752.0
Ca7	25.446	8.11	72.40	783.35	675.8
Ca8	18.231	4.40	54.83	749.58	550.1

4.5. Selection of limestones

Considering the above analysis and knowing that the criteria of discrimination are textural and mineralogical composition of limestones, meaning their purity, a calcium carbonate content close to 100% is required for the remineralization processes. In fact, the mineralogical composition can affect the qualitative and quantitative composition of water during dissolution process for remineralization. Indeed, as this concerns the human consumption, water after treatment must not contain hazardous elements as Os and As. Some such as Li, Na, Mg, Zn, Ni, Fe, are considered micro-nutrients (trace elements) and then good for human health. However, the presence of magnesium slows the dissolution of rock in water. All the analyzed limestones are mainly composed of calcite. ATG-DSC analysis also confirms the predominant presence of calcite which usually decomposes around 790°C. Limestones Ca1, Ca2, Ca3, and Ca4 are marbles collected from the same geological site. But the last tree ones have veins with different color, meaning there is a difference in composition. Limestones Ca5, Ca6, Ca7 and Ca8 bring several other elements than limestone. Therefore, their purity is less than marbles, even if they contain more micro-nutrients. Moreover, their texture leads to believe that their dissolution during remineralization will generate greater production of suspended matter. In the light of the above, marbles have been selected, and, among them, Ca1 corresponds to the rock with greater purity.

Conclusion

The analytical techniques used provided us with valuable insights into the mineral and textural compositions, as well as the thermal characteristics, of various rocks. Chemical analyses of limestone samples sourced from the North, Littoral, and South regions showed that they were primarily made up of calcite. The marbles were found to have similar mineralogical and chemical compositions, with high calcite content and negligible percentages of oxides of Na, K, Mg, Al, Si, Ti, Fe, and P. Although Ca5, Ca6, Ca7, and Ca8 limestones also had high calcite content, they contained several trace elements, some of which posed health risks. The presence of kaolinite and silica was also detected. Their microscopic appearance revealed rhombohedral facies, which is characteristic of calcium carbonates. Therefore, understanding the purity, mineralogical composition, and texture of the rocks is crucial in selecting the appropriate material for a specific purpose. In this study, the rocks' applications were in treating aggressive waters, necessitating high-purity rocks with the lowest amounts of hazardous elements. It is, however, possible to mix various rocks in suitable ratios to increase micronutrient levels while treating water aggressiveness. This study was therefore essential before using limestone rocks for the remineralization water process.

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